

230,91



(19)

Europäisches Patentamt

European Patent Office

Office européen des brevets



(11)

EP 1 048 750 A1

(12)

EUROPEAN PATENT APPLICATION

(43) Date of publication:

02.11.2000 Bulletin 2000/44

(51) Int. Cl.⁷: C23C 30/00

(21) Application number: 00107273.5

(22) Date of filing: 04.04.2000

(84) Designated Contracting States:

AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU
MC NL PT SE

Designated Extension States:

AL LT LV MK RO SI

(30) Priority 26.04.1999 SE 9901485

(71) Applicant SANDVIK AKTIEBOLAG
811 81 Sandviken (SE)

(72) Inventors:

- Blomstedt, Per
756 51 Uppsala (SE)
- Lagerquist, Mikael
194 55 Upplands Väsby (SE)
- Mikus, Marian
127 30 Skärholmen (SE)

(74) Representative:

Tquist, Lennart et al
Sandvik AB
Patent Department
811 81 SANDVIKEN (SE)

(54) Coated cutting tool

(57) The present invention relates to a cutting tool insert comprising a wear resistant coating and a cemented carbide body particularly useful for the machining of cast iron parts by turning, milling or drilling at high speeds. The cemented carbide body consists of WC, 3.5-9 wt % Co and <2 wt-% carbides of Ta, Ti and Nb. It has a core containing finely distributed eta phase islands and an intermediate zone 50-250 µm thick essentially free of eta phase and with nominal Co con-

tent whereby the binder phase in the intermediate zone is present as smaller original islands and larger islands transformed from original eta phase. These latter Co islands therefore have a size and distribution essentially the same as that of the eta phase in the core. There may be present a thin surface zone free of eta phase with a Co content lower than the nominal Co content.

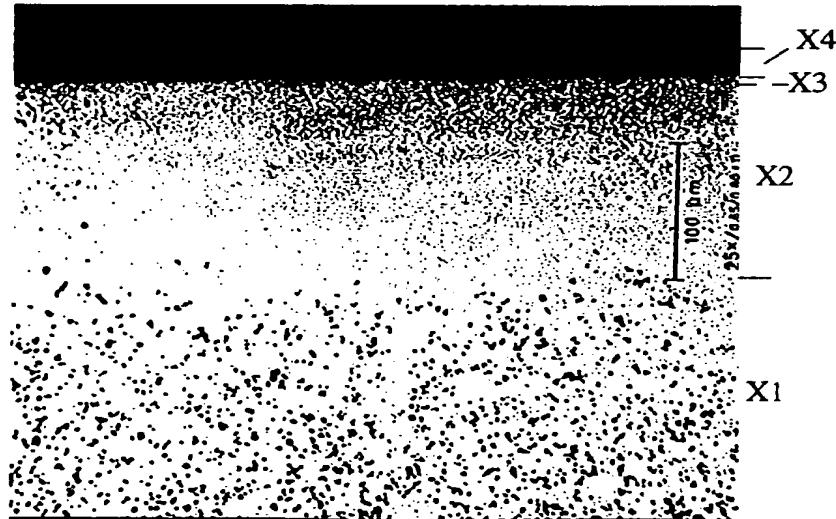


Fig. 1

EP 1 048 750 A1

Description

[0001] The present invention relates to a coated cemented carbide insert particularly useful as a cutting tool for the machining of cast iron at high speeds.

[0002] Cast iron materials may be divided into two main categories, namely grey cast iron and nodular cast iron. From machinability point of view these two materials are quite different. There are also a number of other cast iron materials having intermediate properties in this respect such as the newly developed compact graphite iron.

[0003] Grey cast irons are comparatively easy to machine having graphite flakes well distributed in the microstructure. These flakes provide the formation of short chips and a lubricating effect in the cutting zone. At high cutting speeds the cemented carbide inserts are mainly subjected to abrasive and diffusional wear.

[0004] Nodular cast irons are long chipping materials and their greater deformation resistance leads to a higher temperature level in the cutting zone of the insert. This gives rise to excessive wear due to plastic deformation of the cutting edge by creep.

[0005] US 5,945,207 discloses a coated cutting insert particularly useful for the machining of cast iron parts by turning. It represents prior art for cemented carbide based tools in such applications and recommended cutting speeds when turning grey cast iron and nodular cast iron at a feed of 0.4 mm/rev are 200-300 m/min and 150-200 m/min respectively.

[0006] For the machining of cast iron at higher speeds Si_3N_4 based ceramic tools are normally used. The recommended cutting speeds when using tools of this ceramic material at the same feed as above are 400-700 m/min for turning of grey cast iron and 200-300 m/min of nodular cast iron. However, such tools suffer from brittleness and are more expensive to produce than corresponding coated cemented carbide tools. Therefore, it would be more cost effective if cemented carbide inserts could be used for machining, turning or milling, cast iron components at higher speeds compared to prior art. Further, the use of cemented carbide based inserts instead of ceramic ones decreases the risk of premature rupture and accordingly it increases the possibility to estimate a predictable life of the tools.

[0007] US 4,843,039 teaches how to produce cemented carbide bodies suitable for chip forming machining having a core containing eta phase, M_6C (Co_3W_3C) and/or $M_{12}C$ (Co_6W_6C) embedded in normal alpha (WC) + beta (Co binder phase), said core being surrounded by a surface zone containing alpha and beta phase. The surface zone is free of eta phase and has a lower binder phase content than the nominal content of binder phase in the sintered body. The inner part of the surface zone situated nearest to the core has a content of binder phase greater than the nominal content of binder phase in the sintered body. Thus, the cemented carbide body obtained has a surface zone

with comparatively low cobalt content, i.e. with a high resistance to creep deformation, followed by a zone with high Co content having a high ductility.

[0008] It is an object of this invention to provide a coated cutting tool particularly useful for the machining of cast iron parts by turning, milling or drilling at high speeds.

10 Figure 1 is a micrograph at 40x magnification of the insert cross section showing the microstructural features of a coated insert according to the present invention in which

15 X1 - center of the cemented carbide body containing WC, binder phase and eta phase (M_6C)
X2 - intermediate zone containing WC and binder phase
X3 - surface zone of the cemented carbide body containing WC and a low content of binder phase
X4 - coating.

20 Figure 2 includes micrographs in 1200x magnification - A and -F showing:

25

- A: the microstructure of the intermediate zone (X2), the surface zone (X3) and the coating (X4) of an insert according to the present invention
- 30** - F: microstructure of the same cemented carbide as A with stoichiometric carbon content.

35 **[0009]** According to the invention there is provided a cutting tool insert comprising a wear resistant coating and a cemented carbide body. The cemented carbide body has the composition 3.5-9, preferably 5-8, weight-% Co, <2, preferably <0.5, most preferably 0 weight-% carbides of the metals Ti, Ta and/or Nb and balance WC. The average grain size of the WC in as sintered

40 state is 0.5-4 μm , preferably 1.0-3 μm . The body consists of a core containing eta phase, WC, Co binder phase and possibly gamma phase (cubic carbides), an intermediate zone essentially free of eta phase and a surface zone free of eta phase. The eta phase in the core is finely distributed with a size of 1-15 μm , preferably 3-10 μm , and its content is at least 10 vol-% but at the most 35 vol-%. The amount of eta phase in the core depends on the nominal Co content and at least 20%, preferably 40-80%, of the nominal Co content should be present as Co binder phase and the rest of the Co as eta phase.

45 **[0010]** A surface zone <25 μm thick with Co content somewhat lower than nominal Co content may be present. The intermediate zone is 50-350 μm thick with a Co content essentially equal to the nominal Co content. The binder phase in this zone has a bimodal structure comprising small size and large size Co islands. The large size Co islands are transformed from eta

phase. The small size Co islands comprise islands, which for the most part were present in the structure as Co phase prior to the carburising treatment. The spatial distribution of the large Co islands is essentially the same as that of the eta phase in the core and they are often of an irregular shape with a maximum size somewhat smaller than that of the eta phase in the core.

[0011] In one preferred embodiment the wear resistant coating comprises

- a layer of TiC_xN_y where $x+y=1$, $x>0.3$ and $y>0.3$, with a thickness of 5-10 μm with columnar grains having a diameter of a size <2 μm .

[0012] In another preferred embodiment the wear resistant coating comprises

- a layer of smooth $\alpha-Al_2O_3$ and/or $\kappa-Al_2O_3$ having a grain size of 0.5-2 μm with a thickness of 3-6 μm .

[0013] In a most preferred embodiment the wear resistant coating comprises

- a first, innermost, layer of $TiC_xN_yO_z$ with $x+y+z=1$ and $y>x$ and $z<0.1$ with a thickness of 0.1-2 μm , and with equiaxed grains having a size <0.5 μm
- a second layer of TiC_xN_y where $x+y=1$, $x>0.3$ and $y>0.3$, with a thickness of 5-10 μm with columnar grains having a diameter of a size <2 μm
- a third layer of $TiC_xN_yO_z$ where $x+y+z=1$, $z<0.5$ and $x>y$ with a thickness of 0.1-2 μm and with equiaxed or needle-like grains having a size <0.5 μm
- a fourth layer of smooth $\alpha-Al_2O_3$ having a grain size of 0.5-2 μm with a thickness of 3-6 μm and finally
- an outermost layer of $TiC_xN_yO_z$ where $x+y+z=1$, $z<0.05$ with a thickness of 0.5-3 μm and a grain size <1 μm . Preferably, this outermost layer is missing in at least the edge line so that the Al_2O_3 layer is on top along the cutting edge line and the outer layer of $TiC_xN_yO_z$ is the top layer on the clearance side.

[0014] According to the method of the present invention a cemented carbide body with a composition according to above with substoichiometric carbon content is sintered such that an eta phase containing structure is obtained in which the eta phase is finely distributed with a size of 1-15 μm , preferably 3-10 μm , and a content of at least 10 vol-% but at the most 35 vol-%. The amount of the eta phase in the core depends on the nominal Co content and at least 20%, preferably 40-80%, of the nominal Co content should be present as Co binder phase and the rest of the Co as eta phase. If the carbon content is too close to the stoichiometric carbon content, small amounts of too coarse eta phase are formed. If the carbon content is too low, too much eta phase will be formed. It is within the purview of the skilled artisan to determine by experiments the conditions necessary to obtain the desired microstructure

using his equipment.

[0015] After sintering the cemented carbide is subjected to a gentle recarburisation such that the eta phase in the intermediate and the surface zone is transformed to WC+Co while maintaining, except for the surface zone, essentially the same Co content as that in the eta phase comprising core. The recarburisation is preferably performed at 1250 °C to 1350 °C for 0.5-3 h in a carburising atmosphere such as an H_2+CH_4 -mixture. However, the exact conditions depend strongly upon the equipment used particularly the carbon potential of the furnace. It is within the purview of the skilled artisan to determine by experiments the conditions necessary to obtain the desired microstructure using his equipment.

[0016] The body obtained is coated with wear resistant layers using PVD-, CVD- or MTCVD-methods as known in the art.

[0017] The reason for the observed improvement of inserts according to the invention is probably a unique Co distribution causing increased toughness without loss of plastic deformation resistance so that even at very large feeds no fracture is obtained. A cemented carbide with a Co distribution comprising large Co islands is also possible to obtain using coarse grained WC with a grain size between 4 and 10 μm . However, such cemented carbide will exhibit a high toughness but too low a resistance against plastic deformation during cutting operations at high speed machining. It is believed that the WC skeleton present between large Co islands in inserts according to invention is stronger than that according to prior art. Thus inserts according to the invention have an improved toughness with adequate resistance to plastic deformation during high speed machining.

Example 1

[0018] Coated inserts were made as follows:

A. Cemented carbide cutting tool insert blanks of style CNMA120412-KR for turning of cast iron were pressed from a WC-6% Co powder with 0.18% substoichiometric carbon content and having an average WC grain size of about 2.5 μm . The pressed blanks were then standard sintered at 1450 °C in vacuum with a holding time of 1 hour at the sintering temperature. After conventional surface grinding, edge rounding and cleaning treatments the inserts were resintered under gentle carburising conditions at 1330 °C for 1 hour. The inserts had a microstructure consisting of a core containing about 20 vol-% eta phase with a size of up to 7 μm embedded in the normal WC+Co-structure, followed by an intermediate zone 150 μm thick with a nominal Co content and finally a 10 μm surface zone with a Co content of about 3 wt-%, see Fig 1 and Fig 2 - A. The binder phase in the intermediate

zone had a bimodal structure comprising small sized islands (up to 1.5 μm) and large sized irregular Co islands (up to 5 μm).

The treated inserts were then coated with a 0.5 μm equiaxed $\text{TiC}_{0.1}\text{N}_{0.9}$ layer and an average grain size of 0.2 μm , followed by an 8.0 μm thick $\text{TiC}_{0.4}\text{N}_{0.4}$ layer with columnar grains with an average grain size of 2.5 μm , using MTCVD technique (process temperature 850 $^{\circ}\text{C}$ and CH_3CN as the carbon/nitrogen source). In subsequent process steps during the same coating cycle, a 1 μm thick layer of $\text{TiC}_{0.6}\text{N}_{0.2}\text{O}_{0.2}$ with equiaxed grains and an average grain size of 0.2 μm was deposited followed by a 5.0 μm thick layer of (012)-textured $\alpha\text{-Al}_2\text{O}_3$, with an average grain size of about 1.2 μm , deposited according to conditions given in US 5,654,035. On top of the $\alpha\text{-Al}_2\text{O}_3$ layer, $\text{TiN/TiC/TiN/TiC/TiN}$ was deposited in a multilayer structure with a total coating thickness of 1.5 μm and an average grain size <0.3 μm in each individual layer. Finally the inserts were subjected to a rotary brushing treatment in which the cutting edge lines were smoothed with a nylon brush containing 320 mesh abrasive SiC particles. By this treatment the outer TiN/TiC multilayer was removed along the cutting edge line.

B. Inserts of style CNMA120412-KR with the composition 6.0 weight-% Co and balance WC were sintered in a conventional way at 1410 $^{\circ}\text{C}$ and cooled down to 1200 $^{\circ}\text{C}$ in 0.6 bar H_2 giving inserts with a WC grain size of about 1.3 μm and a binder phase highly alloyed with W and a Co content on the surface corresponding to 6 weight-%. The inserts were then ground, edge roundness treated, cleaned, coated and brushed in the same way as the inserts A. Type B corresponds to prior art according to US 5,945,207.

C. Inserts of style CNMA120412-KR with the composition 3.7 weight-% Co, 2.0 weight-% cubic carbides and balance WC were sintered in a conventional way at 1520 $^{\circ}\text{C}$ giving a WC grain size of about 1.0 μm . The sintered insert blanks were then subjected to identical processes and treatments as insert B.

D. Inserts identical to insert B with the exception that the thicknesses of the TiCN and Al_2O_3 layers in the coating were 4.0 and 10.0 μm respectively.

E. Si_3N_4 ceramic inserts of a commercial grade (Sandvik CC690) and of a style similar to CNMA120412-KR. In order to strengthen the cutting edge to avoid premature rupture a T02520 reinforcement chamfer was ground along the entire edge line.

F. Inserts of style CNMA120412-KR with the composition 6.0 weight-% Co and balance WC were sintered in a conventional way at 1410 $^{\circ}\text{C}$ and cooled down to 1200 $^{\circ}\text{C}$ in 0.6 bar H_2 giving inserts with a WC grain size of about 2.6 μm and a binder

phase highly alloyed with W and a Co content on the surface corresponding to 6 weight-%. The inserts were then ground, edge roundness treated, cleaned, coated and brushed in the same way as the inserts A.

[0019] The inserts were tested in a longitudinal turning operation using coolant. The workpiece consisted of discs of nodular cast iron, SS0727, which were pressed together in order to provide a large amount of cast iron skin, i.e. abrasive wear, and a certain degree of intermittence during each cut. Cutting speed was 400 m/min, feed 0.40 mm/rev and cutting depth 2.0 mm. Three edges per type were tested and the life was determined by any of the following criteria:

- a flank wear (VB) exceeding 0.50 mm,
- rupture, edge fracture,
- excessive wear in the minor cutting edge, or
- excessive wear at the depth of cut.

[0020] The result was as follows:

Insert	Life, number of discs		
	Min.	Mean	Max.
A, (invention)	12.0	12.0	12.0
B	6.0	6.8	7.4
C	4.7	5.9	6.9
D	1.0	4.8	7.5
E	3.0	5.0	6.0
F	5.0	6.0	7.0

[0021] In inserts B, C and D -prior art edge fractures occurred in 10-30% of the tested edges. In insert F plastic deformation of the edge and flaking occurred.

[0022] In a next test, the cutting speed was increased to 750 m/min, other conditions kept constant. The following result was obtained:

Insert	Tool life, number of cuts		
	Min.	Mean	Max.
A (Invention)	2.1	2.7	3.0
B	0.8	2.4	3.0
E	1.5	2.0	2.5
F	1.0	1.5	2.0

[0023] The tests in continuous cut show that the

inserts A have a better performance than prior art in higher productivity machining of nodular cast iron.

[0024] Following these tests, interrupted cut was tried as well. The same cutting conditions were used with cutting speed 650 rpm and feed 0.30 mm/rev. The 5 tool life criterion was fracture of the insert.

Insert	Tool life, number of cuts.		
	Min.	Mean	Max.
A (Invention)	5.0	5.5	6.0
B	4.0	4.0	4.0
E	3.0	3.5	4.0

Example 2

[0025] For further testing the following inserts were prepared and compared to inserts A of Example 1.

G. Inserts of style CNMA120412 having a conventional substrate of WC-6% Co by weight and a WC grain size of 1.0 μm . The coating was similar to the one in type A but the $\alpha\text{-Al}_2\text{O}_3$ layer was somewhat thinner. 1.2 μm .

H. Inserts of style CNMA120412 having the same substrate as type B, see Example 1, and a coating consisting of a 4 μm thick layer of TiAIN deposited by PVD.

I. Inserts of style CNMA120412 having the same substrate as type G and a coating consisting of a 4 μm thick layer of TiCN deposited by PVD.

J. Cemented carbide cutting tool inserts of style CNMA120412 having the same substrate as type G and a coating consisting of a 4 μm thick layer of TiCN/TiN deposited by PVD.

[0026] The test conditions were:

Workpiece: 100% pearlitic compact graphite iron (CGI), cast tube blank $D_y=145$ mm and $D_i=98$ mm.

Cutting speed: 300 m/min

Feed: 0.20 mm/rev.

Cutting depth: 0.5 mm

Tool cutting edge angle: 95°

No coolant

[0027] The life of the inserts was determined as the number of cuts until the flank wear, VB, reached a depth of 0.3 mm. The result so obtained was as follows:

Insert	Tool life, number of cuts
A, (invention)	160
G	110
H	110
I	60
J	30

Example 3

[0028] By using optical image analysis, the micro-structure within the intermediate zone in inserts A was compared to that of similar inserts produced in a conventional way, insert F. The latter inserts consisted of WC-Co cemented carbide having essentially the same WC grain size as insert A, the same nominal Co content as insert A but a stoichiometric carbon content resulting in no eta phase presence. At a magnification of 2000x an area of the size 50x50 μm within the intermediate zone in insert A was analysed using a Quantimet 570, Cambridge Instruments, and compared to the same area within the insert F. The results of the analysis were obtained as an area fraction distribution within 20% steps, between 0 and 100%, as a function of area size. 20 After recalculating the latter areas to a characteristic size corresponding to the diameter of a circle having the same area, the distributions were as follows:

Area fraction (%)	Co-island size μm	
	A, invention	F, prior art
0-20	0-0.5	0-0.35
20-40	0.5-0.8	0.35-0.5
40-60	0.8-1.6	0.5-0.75
60-80	1.6-2.3	0.75-1.0
80-100	2.3-5.0	1.0-2.0

The table shows that insert A, according to invention has much wider Co islands size distribution than that in 50 insert F, prior art.

Claims

1. A cutting tool insert comprising a wear resistant coating and a cemented carbide body characterized in that said cemented carbide body consists of WC, with an average grain size of 0.5-4 μm and 3.5-9 wt-% Co and <2 wt-% carbides of Ta,

Ti and Nb said body further consisting of a core containing finely distributed eta phase islands with a size of 1-15 μm and a content of at least 10 vol-% but at the most 35 vol-%, WC, Co binder phase and possibly gamma phase, an intermediate zone 5-250 μm thick essentially free of eta phase and with nominal Co content and a 0-25 μm thick surface zone free of eta phase with a Co content lower than the nominal Co content whereby the binder phase in the intermediate zone is present as smaller original islands and larger islands transformed from original eta phase and therefore with a size and distribution essentially the same as that of the eta phase in the core.

5

10

15

20

25

25

30

35

40

45

50

55

with average grain size 0.5-4 μm and 3.5-9 wt-% Co and <2 wt-% carbides of Ta, Ti and Nb and with substoichiometric carbon content is sintered such that a body with an eta phase containing structure is obtained in which the eta phase is finely distributed with a size of 1-15 μm and a content of at least 10 vol-% but at the most 35 vol-% whereafter the cemented carbide body is subjected to a gentle recarburisation such that the eta phase in a 50-350 μm wide intermediate zone is transformed to WC+Co without essentially changing its Co content.

2. Cutting insert according to the preceding claim characterized in that said coating comprises a layer of TiC_xN_y where $x+y=1$, $x>0.3$ and $y>0.3$, with a thickness of 5-10 μm with columnar grains having a diameter of a size <2 μm . 20
3. Cutting insert according to claim 1 characterized in that said coating comprises a layer of smooth $\alpha\text{-Al}_2\text{O}_3$ and/or $\kappa\text{-Al}_2\text{O}_3$ having a grain size of 0.5-2 μm with a thickness of 3-6 μm . 25
4. Cutting insert according to claim 1 characterized in that said coating comprises
 - a first, innermost, layer of $\text{TiC}_x\text{N}_y\text{O}_z$ with $x+y+z=1$ and $y>x$ and $z<0.1$ with a thickness of 0.1-2 μm , and with equiaxed grains having a size <0.5 μm ; 30
 - a second layer of TiC_xN_y where $x+y=1$, $x>0.3$ and $y>0.3$, with a thickness of 5-10 μm with columnar grains having a diameter of a size <2 μm
 - a third layer of $\text{TiC}_x\text{N}_y\text{O}_z$ where $x+y+z=1$, $z<0.5$ and $x>y$ with a thickness of 0.1-2 μm and with equiaxed or needle-like grains having a size <0.5 μm ; 40
 - a fourth layer of smooth $\alpha\text{-Al}_2\text{O}_3$ having a grain size of 0.5-2 μm with a thickness of 3-6 μm ; and finally
 - an outermost layer of $\text{TiC}_x\text{N}_y\text{O}_z$ where $x+y+z=1$, $z<0.05$ with a thickness of 0.5-3 μm and a grain size <1 μm . 45
5. Cutting insert according to claim 4 characterized in that said outermost layer is missing in at least the edge line so that the Al_2O_3 layer is on top along the cutting edge line and the outer layer of $\text{TiC}_x\text{N}_y\text{O}_z$ is the top layer on the clearance side. 50
6. Method of making a cutting insert comprising a cemented carbide body and a coating characterized in that a cemented carbide with WC

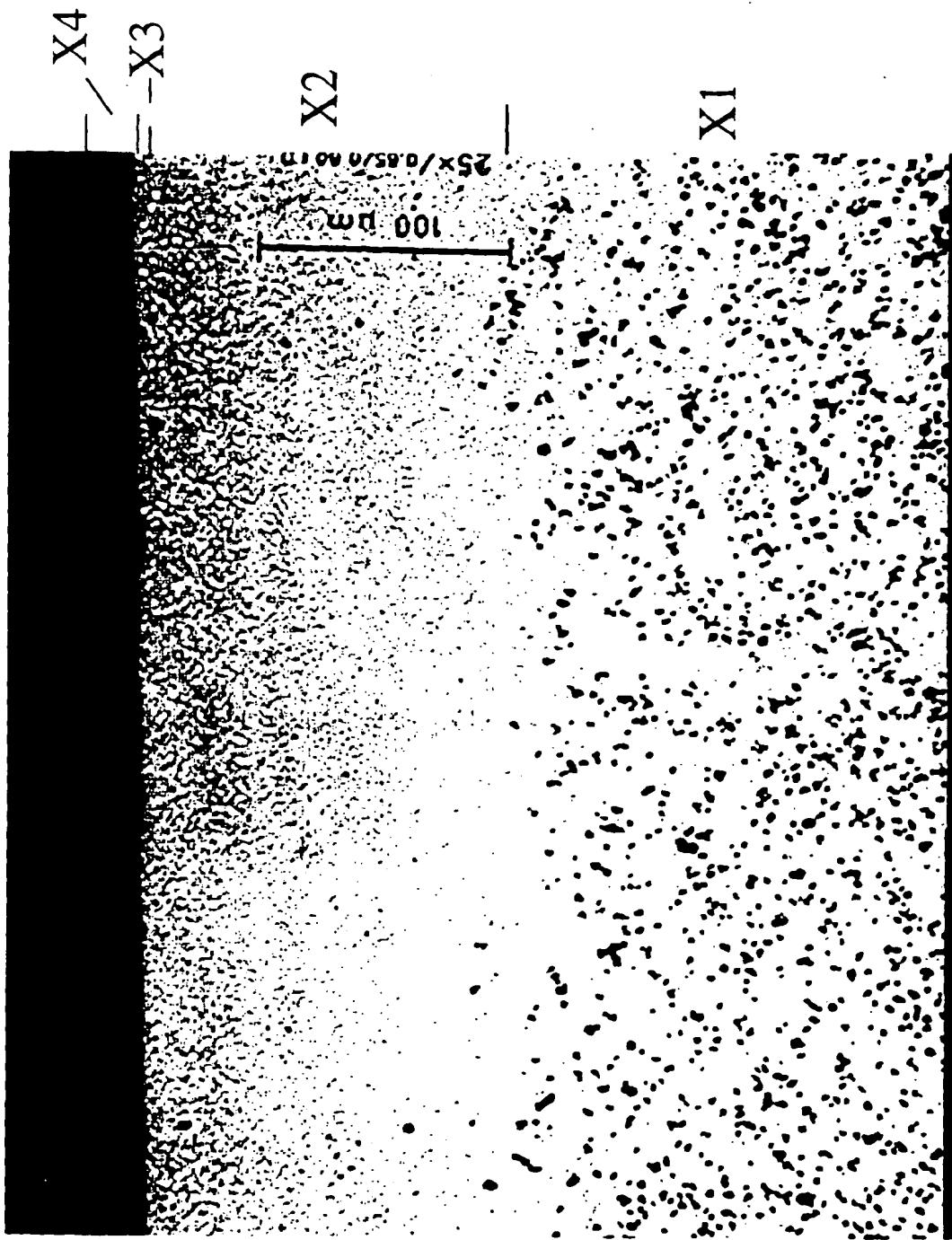
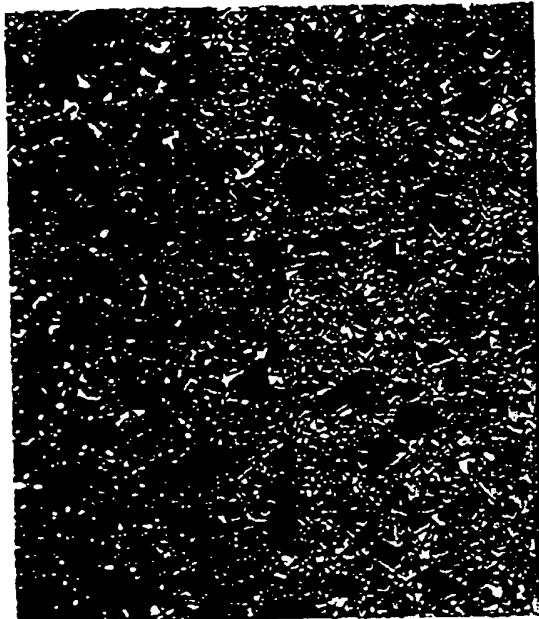
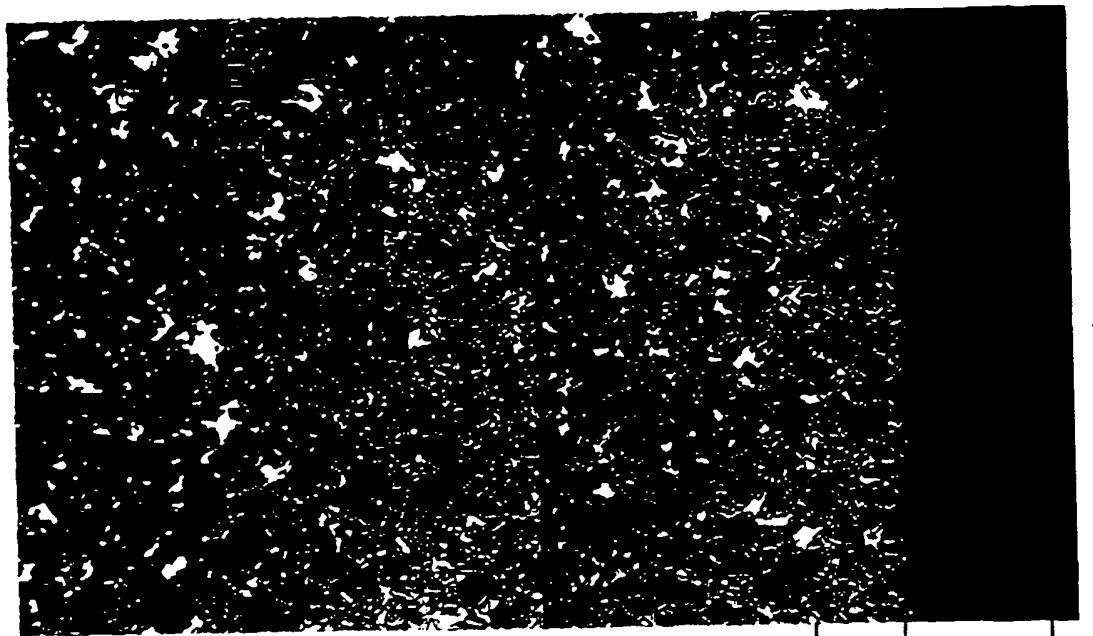


Fig. 1

BEST AVAILABLE COPY



F



A

X2

X3

X4

BEST AVAILABLE COPY



EUROPEAN SEARCH REPORT

Application Number

DOCUMENTS CONSIDERED TO BE RELEVANT			EP 00107273.5						
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 7)						
X	WO 98/02598 A (SANDVIK AB) 22 January 1998, the whole document. --	1-6	C23C30/00						
X	WO 98/10119 A (SANDVIK AKTIEBOLAG) 12 March 1998, the whole document. --	1-6							
X	EP 0753603 A (SANDVIK AKTIEBOLAG) 15 January 1997, the whole document. -----	1-6							
TECHNICAL FIELDS SEARCHED (Int. Cl. 7)									
C23C									
<p>The present search report has been drawn up for all claims</p> <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 33%;">Place of search</td> <td style="width: 33%;">Date of completion of the search</td> <td style="width: 34%;">Examiner</td> </tr> <tr> <td>VIENNA</td> <td>06-06-2000</td> <td>BECK</td> </tr> </table>				Place of search	Date of completion of the search	Examiner	VIENNA	06-06-2000	BECK
Place of search	Date of completion of the search	Examiner							
VIENNA	06-06-2000	BECK							
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document							

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO. EP 00107273.5**

This annex lists the patent family members relating to the patent documents cited in the above-mentioned search report. The members are as contained in the EP0095 COMPACT file on 01.01.1998. The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO A1 9802598	22-01-1998	EP A1 910685 SE A0 9602753 SE A 9602753 SE C2 510778 US A 5942318	26-04-1999 11-07-1998 12-01-1998 21-04-1999 24-08-1999
WO A1 9810119	12-03-1998	CN A 1229442 EP A1 9803065 SE A0 9803264 SE A 9803264 SE C2 509560 US A 5945207	22-09-1999 03-11-1998 06-09-1998 07-02-1999 09-02-1999 31-08-1999
EP A2 753603	15-01-1997	AT E 188751 CN A 1142421 DE CO 69506109 DE T2 69506109 EP A3 753603 EP B1 753603 IL A0 118791 IL A1 118791 JP A2 9029512 SE A0 9502640 SE A 9502640 US A 5863640	15-01-2000 12-02-1997 17-02-2000 21-05-2000 14-05-1997 12-01-2000 31-10-1998 26-01-1999 04-02-1997 14-07-1995 15-01-1997 26-01-1999

For more details about this annex see Official Journal of the European Patent Office, No. 12/82.

BEST AVAILABLE COPY